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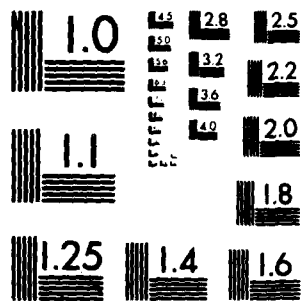
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## Electrical Engineering Department

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Investigation of Cold Cathodes  
for  
Long Life CO<sub>2</sub> Waveguide Lasers

Final Report  
for Contract F49620-79-C-0076

Submitted to:

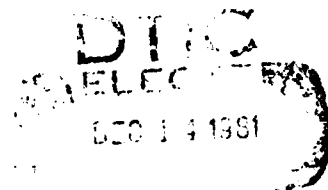
Dr. H. Schlossberg

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by

U. Hochuli

November 1981



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Abstract

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## Introduction

The cold cathode technology developed earlier for the low pressure CW CO<sub>2</sub> laser gives useful guidelines for the selection of proper cathode materials. Using these guidelines for the CO<sub>2</sub> waveguide laser, working at pressures around 120 Torr, still yields cathodes that allow maintenance of reasonable gas mixture compositions over periods in excess of 10<sup>4</sup> hours. The main difficulties with most of these cathodes arise from loose, flaky, sputtering deposits and from mirror contamination. Elevating the cathode temperature can reduce sputtering and prevent these flaky deposits. Unfortunately, this leads to a non-viable engineering solution due to the additional heater required.

Careful observations on the more than 50 different cathodes being tested indicate that semiconducting, sintered NiO is one of the better cathode materials. Such cathodes were actually used to generate the life test data with two of the BeO laser structures shown in Figure 1. Both of these lasers used three cathode type electrodes, one located 5/8" from each bore end and one near the bore center. Laser No 1 used both end electrodes as 2mA cathodes and the center electrode as the common 4mA anode. Laser No 2 worked with the end electrodes as 2mA anodes and used the center electrode as a common 4mA cathode. From the discharge tube results we know that the 4mA cathode is prone to considerably heavier sputtering than the 2mA cathode. Both lasers had indium "O" ring and indium film seals developed by us 11 years ago. All the seals on Laser No 1 were perfect but Laser No 2 had



a very small crack in the BeO body which was sealed with "Black Seal" liquid.

#### Life Test Results

The output power vs time curves for these two lasers are shown in Figures 2 and 3. These curves indicate a half power life of about 8000 hours for laser No 1 and about 4300 hours for laser No 2. Our results are quite an improvement when compared to the 750 to 1500 hour results we achieved a year ago. Probably an even longer life could have been achieved if the gas supplies for both lasers had not been reduced from 80cc to 35cc between 2500 and 3000 hours.

#### Failure Analysis:

A variety of experiments were performed on the two lasers in order to determine the possible failure mechanisms. Details of these experiments are given below:

##### a) Laser No 1

As Figure 2 shows the power output of this laser decreased from 1.54W to 0.7W after 9500 hours. Evacuation to 1 Torr and refilling restored the power to only 0.86W. Vacuum pumping and 100°C heating for two days, followed by refilling, restored the output power to 0.9W. Our laser structures use external windows (uncoated ZnSe, tilted 7° to avoid retroreflections) as vacuum seals behind the internal mirrors. Power supply failure over one weekend lowered the laser body temperature to the 15°C cooling water temperature.

Humidity had condensed on these windows and together with the dust and the grime formed water spots. A cotton swab and alcohol treatment could not remove these spots. At the time we thought that this visual degradation would not absorb much power at  $10.6\mu$ . Repolishing these windows, on the laser, with Linde type A and B  $Al_2O_3$  polishing compound improved the laser power output to 1.34W. Replacement of two additional mirrors (front surface Al coated, used for deflection of power from both laser ends onto power meter) further increased the power meter reading to 1.4W. This value is within 10% of the original 1.54W and is an indication that no serious mirror damage had occurred. Removal of the laser mirrors revealed the central 1.5mm area either by breathing on it or by noticing a very faint greenish hue through observation of the surface at a glancing angle. Very few specks of black NiO with diameters of 10 to 30 microns could be seen in the BeO cathode cavities. The  $Al_2O_3$  sputter shields also had a light black color in the central hole and the color gradually thinned out into a very light greenish hue over most of its front surface. This greenish hue did not seem to extend into the cathode cavity of the BeO body.

Auger spectroscopic analysis, performed by the National Security Agency, showed about equal peaks of Ni and C plus an excess of O for the cathode sputtershield deposits and a carbon layer on the mirror spots. One mirror spot had a  $50A^\circ$  thick carbon layer while the other mirror spot had a much thinner layer which may have been the result of  $CO_2$  adsorption.

No trace of chlorine was found on either mirror. These results are strange because for a laser which is symmetrical with respect to both mirrors we expect similar results for both mirrors. Optically we did indeed see similar spots of faint discoloration for both mirrors. We believe that the heavier, or even both carbon deposits resulted during the gas refilling operation that took place after 9500 hours. We refilled the laser slowly but even then we may have stirred up carbon deposits in the bore and swept them to the mirrors. Since the gas inlet port was asymmetrically located behind one of the cathodes it could explain the different thicknesses of the mirror deposits. Later, this carbon dust may have absorbed enough laser power to bake on to the mirror surface. So far we do not think that we have a good explanation for the optically visible mirror spots and we may have to analyze these areas again. It may also turn out that Auger surface analysis can not explain these discolorations which may be the result of UV exposure. We have in the past observed permanent color changes of dielectrically coated ZnSe mirrors as a result of simple heating to perhaps 200 to 300°C in air.

b) Laser No 2

Figure 3 indicates a power drop from an initial 1.5W to 0.54W after 4750 hours. Increasing the gas volume again to 80cc, by reconnecting the laser to the same gas supply

that was used for the first 2900 hours, raised the output power to 1.2W. Pumping down to 1 Torr and refilling the laser with new gas increased its power output to 1.4W compared to 1.5W for the new laser. This indicates that no serious mirror damage had occurred. The laser was then carefully evacuated with the Vac-Ion pump and outgassed for two days at 100°C. Refilling and measuring the power output gave 1.39W or essentially the same as before. 1300 hours later this output was down to 0.68W, almost as before. Leak testing revealed a small leak through the once repaired crack in the BeO structure. This leak was most likely caused by the previous 100°C heating cycle.

Opening the structure revealed a crater like, metallic looking, material build-up around the center hole in the cathode sputter shield. This deposit turned out to be Ni originating from the sputtered, reduced NiO of the cathode. The cathode cavity in the BeO body contained about 20 flakes in sizes ranging from 10 to 50 microns in diameter. About half of them appeared metallic and were magnetic and the rest looked like black NiO. X-ray fluorescence analysis confirmed Ni as the dominant composition of the flakes.

Inspection of the mirrors under a microscope revealed no surface degradation. Breathing on the mirrors enabled us to distinguish the central area opposite to the 1.5mm bore. X-ray fluorescence analysis also distinguished this area but did not reveal a different composition from the rest of the

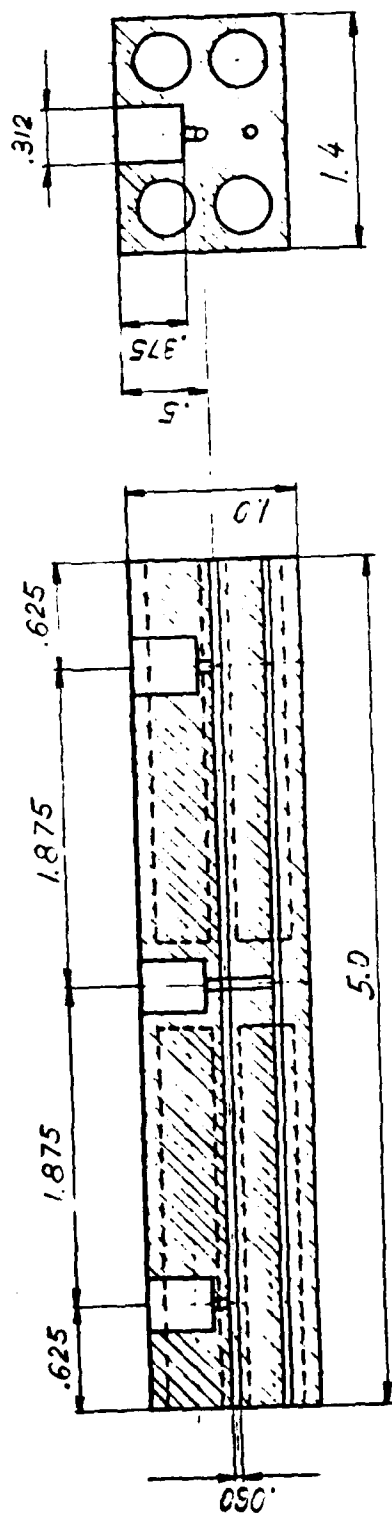
mirror. Auger spectroscopy, performed at the Naval Research Laboratory, showed an excess of chlorine and oxygen in the central mirror area. The excess oxygen most likely resulted from either the pure oxygen discharge used to clean the laser before the actual filling took place or from the exposure to the laser discharge that always contains oxygen from dissociated  $\text{CO}_2$ . However, the presence of chlorine is more difficult to explain. One possible chlorine source may be the indium "O" rings. These "O" rings were cleaned in 50% HCl followed by ultrasonic cleaning in distilled  $\text{H}_2\text{O}$  and alcohol. Any formation of  $\text{InCl}$ ,  $\text{InCl}_2$  and  $\text{InCl}_3$  should easily be dissolved in  $\text{H}_2\text{O}$  and washed away. Other chlorine sources may be: the 0.0005% Cl present in the nickelous nitrate used as the basis material for our NiO cathodes and/or chlorine presence in the 5% binder used in the  $\text{Al}_2\text{O}_3$  sputtershield ceramic.

### Conclusions

Within the past year we have extended the life of 1.5W  $\text{CO}_2$  waveguide laser structures from a low 500 to 1500 hour range into the 4000 to 8000 hour domain. Laboratory failure seems to have been due to  $\text{CO}_2$  depletion of the gas supply. Gas volumes started at 80cc and were cut back to 35cc after 2500 to 3000 hours where they remained for the rest of the life tests.

It should be carefully pointed out that these are laboratory results. In actual field use, absolutely none of

the observed loose flakes could be tolerated. We must also mention that the recorded power output corresponds to the most powerful transition in the laser's signature. For the duration of the laser life the output drifted around an average value that was about one half of its peak. Under these conditions we have observed no serious degradation of the dielectric coated ZnSe mirrors. However, we do not know if this statement would still be correct if our lasers had been continuously tuned for maximum output power.



LASER STRUCTURE  
(BeO)

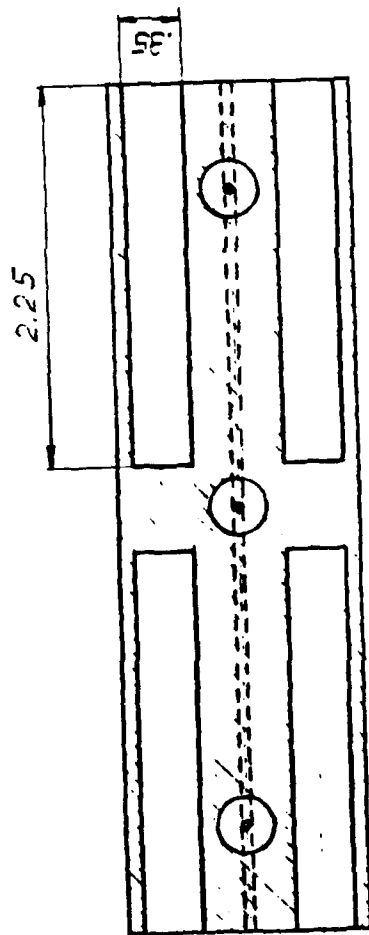


Fig. 1

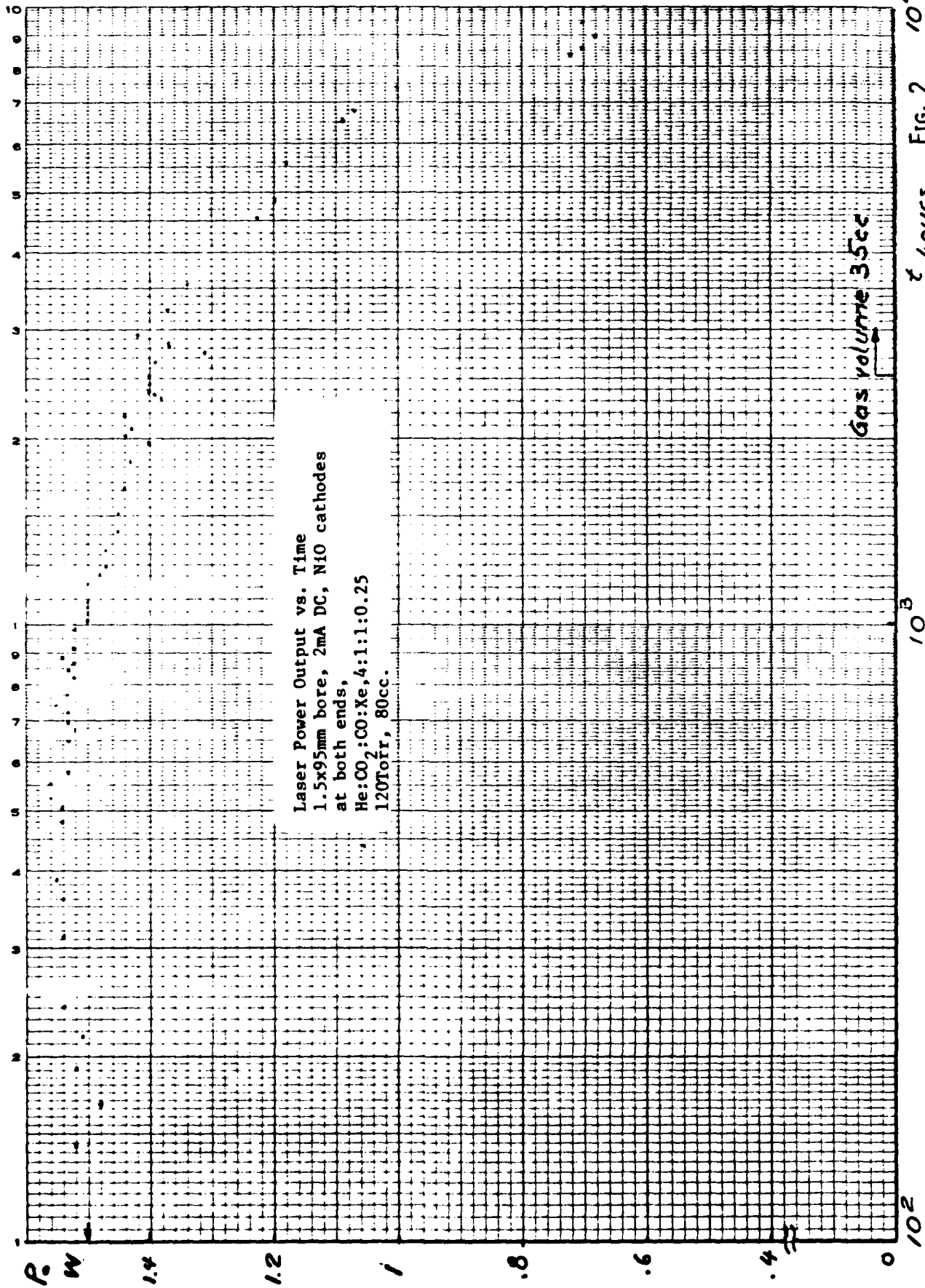


FIG. 2 10<sup>4</sup>



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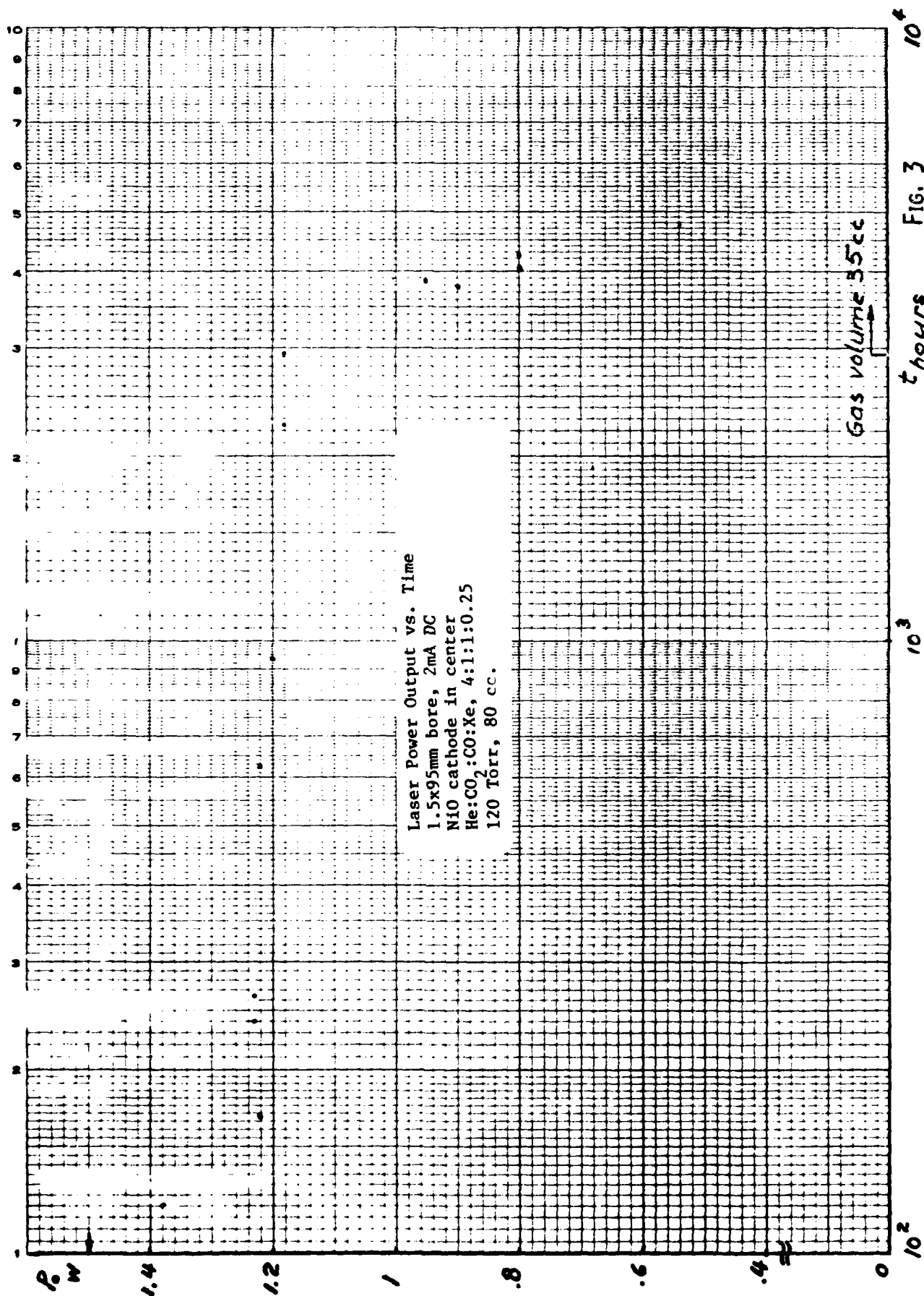
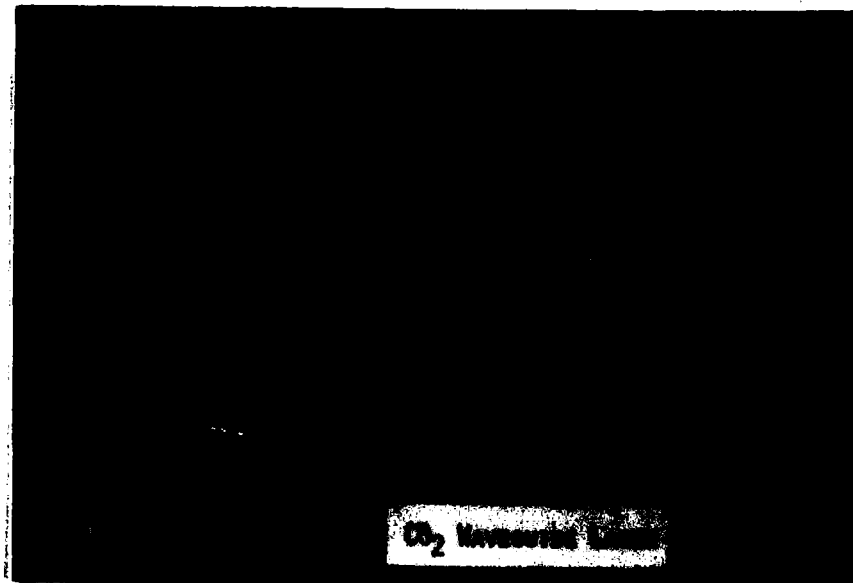
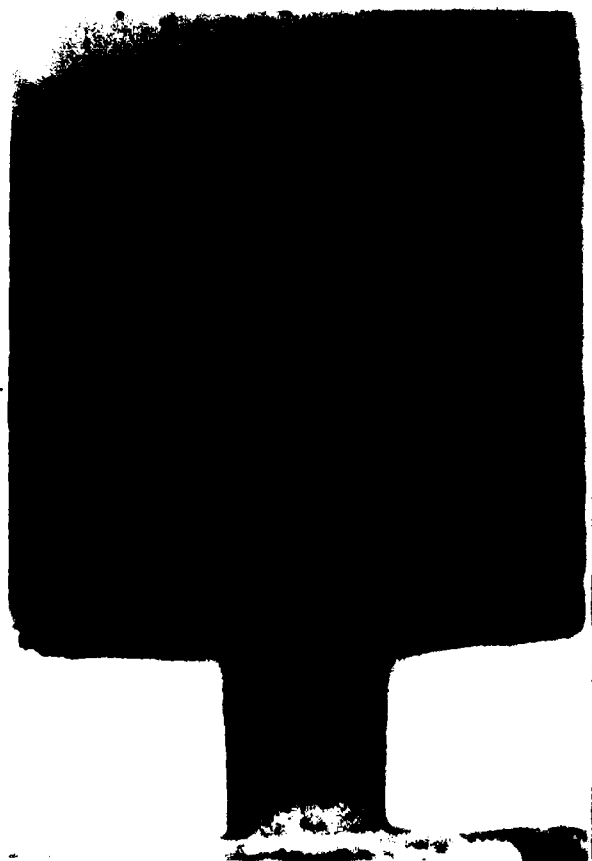
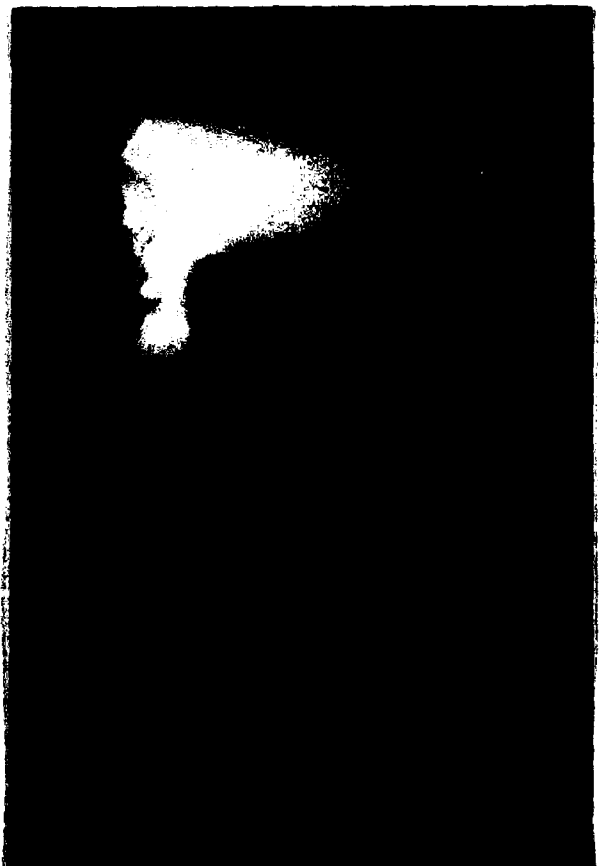
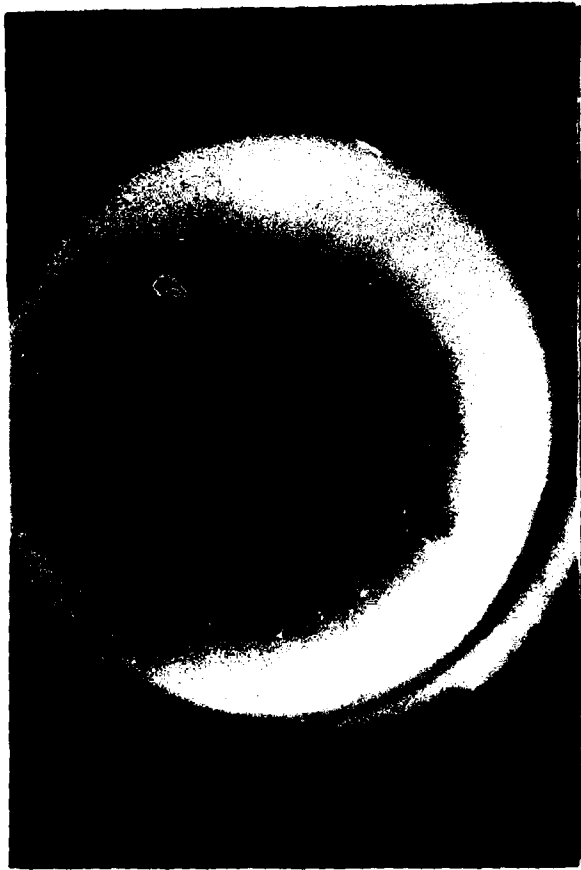


FIG. 3

# FAILURE ANALYSIS







Laser No 1

2 mA cathodes at each bore end, common 4 mA anode in center. All parts shown have served 9500 hours

Enlarged NiO cathode cylinder with sputtering deposits

NiO anode cylinder on top, NiO cathode cylinder below. Both holes have 1.5mm ID and are 1.5mm deep.

NiO cathode cylinder, held in  $Al_2O_3$  sputtershield with transparent Epoxy resin before cutting in half and polishing. Cathode hole erosions as deep as 0.14mm are clearly visible. Dark sputtering products line only the inner part of the sputtershield hole.

The small, bright spots are trapped air bubbles in the Epoxy resin and should be disregarded.

Laser mirror with spot. The color film very much enhanced the spot which is barely visible to the naked eye.

Laser No 2

2 mA anodes at both bore ends, common 4 mA cathode in center. All parts shown have served for 6300 hours.

$\text{Al}_2\text{O}_3$  sputtershield with sputtered NiO about half of it reduced to pure Ni. Sputtershield of laser No 1 shows no flakes, only slight discoloration, darker around hole and fading out towards the edge.

Enlarged hole section of sputtershield with flaky, sputtered deposits.

Deposits on lighter colored areas have flaked off.

Cathode-and Anode NiO cylinders.

Cathode cylinder was split to show the inside of the cathode hole in the picture to the right.

Split NiO cathode cylinder, placed in split  $\text{Al}_2\text{O}_3$  sputtershield.

Hole in sputtershield is uniformly lined with dark sputtering products.

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